

Dissipation of Nicosulfuron and Rimsulfuron in Surface Soil

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Field and soil fortification studies were conducted to evaluate the half-lives (DT₅₀) of nicosulfuron and rimsulfuron in a Sequatchie silt loam surface soil. The dissipation of each herbicide was also evaluated with the two compounds applied simultaneously, which is a typical application method used in corn production. Field studies in two years indicated that both herbicides alone and in mixture disappeared quickly, with all DT₅₀ < 6 days. Environmental conditions including warm, moist soil, and a soil pH of 5.7 encouraged rapid herbicide dissipation. Rapid degradation was observed under laboratory conditions using this same soil, with all DT₅₀ < 3 days. This research indicated minimal risk of carry-over to subsequent rotational crops and minimal residual weed control from these herbicides when applied to a silt loam soil under ambient climatic conditions in Tennessee.

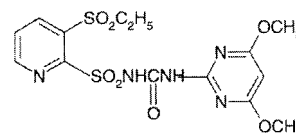
KEYWORDS: Solid phase extraction; sulfonylurea; HPLC; persistence

INTRODUCTION

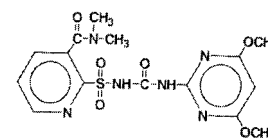
Pesticide dissipation from surface soil is influenced by many factors including soil pH, climate, temperature, and rainfall (1). The optimum rate of loss of a soil-applied herbicide is a compromise between greater persistence, and thus more residual efficacy, and the desire for rapid degradation to minimize environmental risk.

Sulfonylurea herbicides typically degrade via a combination of bridge hydrolysis by a chemical route and microbial degradation (2, 3). Chemical hydrolysis cleaves the sulfonylurea bridge, resulting in sulfonamide and *s*-triazine derivatives (4). A much less common degradative pathway involves rearrangement via bridge contraction. Sulfonylurea dissipation is influenced not only by the chemical structure of the parent herbicide but also by soil organic matter, soil pH, and environmental conditions resulting from varying moisture and temperature combinations (2).

Rimsulfuron [N-[(4,6-dimethoxy-2-pyrimidinyl)amino]carbonyl]-3-ethylsulfonyl-2-pyridinesulfonamide] has two primary uses in agricultural production systems (Figure 1). It is sold as a single-entity product for use in potato weed control systems (5, 6) and as a component in package mixtures for weed control in corn (7, 8). The fate of [¹⁴C]rimsulfuron in soil environments indicated that hydrolysis followed first-order kinetics, was pH dependent, and was more rapid at higher temperature (9). Soil metabolism studies also showed rapid decomposition by bridge contraction with half-lives of 25 days (laboratory) and 5.7 days (field). Minimal mobility was observed under field conditions. In microbially active soil, the mean half-life for rimsulfuron was 7.5 days in an Italian soil, with no effect on soil microbial activities at the concentrations used in agricultural practice (10).



Rimsulfuron



Nicosulfuron

Figure 1. Chemical structures for rimsulfuron and nicosulfuron.

Neither rimsulfuron nor its metabolites were detected at soil depths lower than 8 cm, and rimsulfuron half-life in the 0–8 cm surface layer was 14 days in a Belgium soil (11). Rimsulfuron half-life ranged from 5 to 6 days in an Italian soil, and a recropping interval of 2–3 weeks proved to be adequate to avoid injury to any crops (12). Rimsulfuron degradation under controlled conditions in a silt loam soil showed half-lives from 25 to 1.5 days, corresponding to soil temperatures of 5–35 °C at 80% field capacity (13). In a study using alluvial soil columns, the risk of ground water contamination by rimsulfuron seemed “very low”, as it was rapidly degraded under dynamic conditions (14). It was also reported that rimsulfuron would not create problems of aquifer pollution, due to its fast hydrolysis in a wide range of pH and temperature conditions (15).

Nicosulfuron [2-[[[(4,6-dimethoxy-2-pyrimidinyl)amino]carbonyl]amino]sulfonyl]-N,N-dimethyl-3-pyridinecarboxamide] is applied after corn has emerged for control of grass species

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(Figure 1) (16). Few published citations exist on the behavior of nicosulfuron in soils that are typically used for corn production. Research suggested nicosulfuron would not be highly mobile in soils with a 2:1 clay mineralogy, indicating that in such soils its potential to leach to ground water may be lower than that of other sulfonylurea herbicides (17, 18). Under a simulated rainfall test system, nicosulfuron did not move from the treated area, and thus would not pose a significant threat to the environment (19).

Previous research implied that rimsulfuron and nicosulfuron would probably not be persistent in the environment in surface or ground water systems (15). The primary interest in this research was to determine the persistence of each herbicide as an indication of residual weed control and if any potential to injure rotational crops existed. There is no refereed publication reporting the fate of these herbicides under field conditions in the mid-south area in the United States, which is a major use area for these herbicides. Additionally, several modifications were needed to enable the sample preparation procedure to adequately remove interfering components yet still recover the herbicide(s). The objectives of this study were (1) to optimize a soil extraction/cleanup/analysis regime for a soil in the typical use area of rimsulfuron plus nicosulfuron, (2) to determine the half-lives of nicosulfuron and rimsulfuron under field conditions in Tennessee and laboratory conditions in a medium-textured soil, and (3) to examine the effect of the two sulfonylurea herbicides nicosulfuron and rimsulfuron applied simultaneously on their respective dissipation rates, because this corresponds to a typical use pattern of these herbicides.

MATERIALS AND METHODS

Field Study. Field plots were established in 1997 and 1998 on a Sequatchie silt loam (fine-loamy, siliceous, thermic Humic Hapudult). The pH of this soil was 5.7, cation exchange capacity was 7.0 cmol g⁻¹, organic matter content was 1.3%, and sand/silt/clay content was 27/59/14%, respectively. Soils were characterized using standard assay procedures in our laboratory. Plots were established on bare, tilled ground and were 3 m × 15 m. The study consisted of four treatments, including nicosulfuron at 46 g of active ingredient (ai) ha⁻¹, rimsulfuron at 46 g of ai ha⁻¹, a combination of nicosulfuron at 46 g of ai ha⁻¹ + rimsulfuron at 46 g of ai ha⁻¹, and a nontreated control. Treatments were applied using a CO₂-backpack sprayer at 140 L ha⁻¹ at 240 kPa on June 30 of each year. Field applications were made to simulate a typical timing for these postemergence herbicides, and commercial herbicide formulations were used (7, 8, 16). The study utilized a randomized complete block design with four replicates.

Surface soil samples were taken randomly within plots using a hand-held 8 cm core sampler. Two cores were taken per plot and mixed to form a composite sample for that individual plot. The sampler was cleaned between plots, and other appropriate sampling and experimental considerations were taken (20). The samples were put into plastic bags and immediately frozen at -10 °C until analysis. In 1997, samples were taken to a depth of 0–10 cm at 0, 2, 4, 7, 11, 14, 18, 31, and 60 days after treatment (DAT). Samples were taken to a depth of 0–8 cm in 1998 at 0, 1, 2, 3, 4, 7, 11, 14, 18, and 31 DAT. Samples taken 60 DAT in 1997 and 31 DAT in 1998 were not extracted, because chemical analysis indicated that herbicide concentrations had decreased below the method's limit of detection.

Laboratory Study. Moist soil (70% field capacity) was taken from the field study untreated area and was placed in low-density polyethylene (LDPE) bottles (50 g dry weight basis) and fortified with herbicides, which were dissolved in methanol. The total amount of methanol added was 1 mL, and it was allowed to partially evaporate (4 h) prior to sample mixing. Treatments consisted of nicosulfuron alone, rimsulfuron alone, nicosulfuron plus rimsulfuron, and a nontreated control (all initial concentrations were 50.0 ng g⁻¹, soil basis).

Samples were then placed in an incubator at 30 °C and removed at 0, 1, 2, 3, and 7 DAT. Samples were frozen at -10 °C until analysis. The experiment was conducted twice with three replications, and data were combined for the two studies due to lack of interactions in the statistical analysis.

Herbicide Extraction. Extraction methods were modified from those of Powley and de Bernard (21). Because there were several substantial changes to this method, our entire analysis sequence is presented. Nicosulfuron and rimsulfuron were simultaneously extracted and analyzed using the same methodology. Soil samples from the field study were thawed and then thoroughly homogenized. Moist soil (50 g) was placed into a 250 mL LDPE bottle. The soil was extracted with 100 mL of aqueous ammonium carbonate (0.1 M)/acetone solution (90:10 v/v) by agitation on a reciprocating shaker for 20 min. Samples were centrifuged in a refrigerated centrifuge (4 °C) at 5000 rpm for 45 min. The supernatant was decanted through glass wool into a 250 mL Erlenmeyer flask, and 100 mL of extracting solution was added to the soil to repeat the extraction procedure. The supernatants were combined. Extraction was completed 1 day before cleanup, and samples were stored in a refrigerator at 4 °C overnight. A 120 mL aliquot of the supernatant was placed in a clean LDPE bottle and the pH adjusted to between 3.0 and 3.5 with 85% aqueous phosphoric acid to protonate the herbicide molecule before it passed through the solid phase extraction cartridge.

Sample Cleanup. Solid phase extraction, while under negative pressure at 110 kPa (±15 kPa), was completed using a vacuum extraction manifold to hold cartridges. The C₁₈ (2 g per 12 mL) cartridge (Varian part no. 1225-6008, Sugarland, TX) for the first cleanup was preconditioned with 5 mL of methanol followed by 10 mL of water. The sample extract was slowly passed (5 mL min⁻¹) through the individual column, allowing individual droplets to form at the column base. After all of the supernatant had passed through it, the cartridge was rinsed with 5 mL of water. A graduated 13 mL centrifuge tube was placed under each cartridge. The cartridge was then eluted with 10 mL of 0.1% glacial acetic acid in ethyl acetate. Samples were placed in a 35 °C water bath and dried using a stream of nitrogen gas. Samples were reconstituted in 1 mL of acetone and mixed well using a vortex mixer and sonicated for 5 min. The samples were placed in the water bath and dried a second time. This process removed any excess water. Ethyl acetate (2 mL) was added to reconstitute the sample, which was mixed with a vortex mixer and sonicated for 5 min. Then, 10 mL of hexane was added.

A silica cartridge (1 g per 6 mL, Varian part no. 1225-6012) was used for the second cleanup due to the high affinity sulfonylurea herbicides have for silica. The silica cartridge was preconditioned with 5 mL of ethyl acetate followed by 5 mL of hexane/ethyl acetate (80:20, v/v). The sample extract was then passed through the column. The tube was rinsed with an additional 5 mL of hexane/ethyl acetate mixture, and the rinsate was passed through the column. The herbicide was then eluted from the column with 15 mL of 0.5% glacial acetic acid in acetone into a centrifuge tube. The sample extract was dried in a 35 °C water bath under nitrogen gas.

The sample was reconstituted in 1 mL of acetone, vortexed, and sonicated for 5 min; then 1 mL of potassium phosphate buffer at pH 6.2 was added and the sample reduced to <1 mL under a nitrogen stream. Samples were brought to 1 mL with pH 6.2 potassium phosphate buffer and passed through a 0.45 mm syringe filter into a 4 mL vial. The sample was then brought to 2.0 mL in pH 6.2 potassium phosphate buffer, which was the same as a mobile phase component, and then injected for analysis.

Herbicide Analysis. Samples were analyzed using reversed-phase high-performance liquid chromatography (RP-HPLC). The column was a Zorbax 4.6 × 250 mm SB-phenyl analytical column with a Zorbax SB-phenyl guard cartridge (Agilent Technologies, Agilent.com). All solvents were of HPLC grade and consisted of acetonitrile and 0.30 mM potassium phosphate buffer at either pH 2.7 or 6.2. Buffered mobile phases were adjusted to the proper pH by using 85% aqueous phosphoric acid for the pH 2.7 solution and 10% ammonium hydroxide for the pH 6.2 solution. Mobile phase flow rate was constant at 1.0 mL min⁻¹. A description of the gradient program used is in Table 1, and all changes were made using a linear gradient. The concept of the

Table 1. Mobile Phase Flow Gradient Table for HPLC Analysis of Nicosulfuron and Rimsulfuron

time, min	acetonitrile, %	30 mM phosphate buffer, %	
		pH 2.7	pH 6.5
initial	23	72	5
10.05	10	5	85
15.00	10	5	85
30.00	30	5	65
30.05	50	25	25
40.00	23	72	5
60.00	23	72	5

Table 2. First-Order Dissipation Rate Constants (*k*), Calculated Half-Lives (DT₅₀), and 95% Confidence Intervals (CI) for Herbicides in Surface Soil from Field Experiments

year	treatment	r ²	k, day ⁻¹	DT ₅₀ , days	CI, days
1997	nicosulfuron alone	0.92	0.13	5.3	3.6–9.7
	nicosulfuron mix	0.89	0.17	4.2	2.7–9.4
	rimsulfuron alone	0.95	0.22	3.1	2.3–5.4
	rimsulfuron mix	0.85	0.20	3.5	2.0–13.9
1998	nicosulfuron alone	0.91	0.54	1.3	0.8–3.5
	nicosulfuron mix	0.85	0.32	2.2	1.63–6.6
	rimsulfuron alone	0.83	0.56	1.2	0.7–3.8
	rimsulfuron mix	0.91	0.32	2.2	1.4–4.2

gradient program was to load and concentrate the herbicide at the column head due to the initially low pH. The water soluble components were eluted due to the largely aqueous nature of the initial mobile phase. Nicosulfuron and rimsulfuron were eluted from the column by increasing the organic solvent concentration and increasing the pH, thus increasing the herbicide solubility. A variable wavelength absorbance detector was operated at 245 nm for determination of nicosulfuron and rimsulfuron. Data were captured using a computer data system that integrated the peak area of the chromatogram. Recoveries for nicosulfuron and rimsulfuron were both 70 ± 5%. Herbicide concentrations were determined using an external standard technique, and concentrations were corrected for recovery, antecedent soil water content, and concentration/dilution steps in the method. Analytical standards of nicosulfuron (95%) and rimsulfuron (99%) were used. Samples were processed in batches of eight, and experimental replication identity was maintained throughout analysis. A fortified control sample (10 ng g⁻¹, soil basis) was included with each extraction run as a measure of herbicide recovery. The retention times were 43 and 30 min for rimsulfuron and nicosulfuron, respectively. A conservative lower limit of detection was 2 ng g⁻¹, soil basis.

Data were fit to first-order kinetics using an SAS nonlinear (NLIN) regression procedure (22). The herbicide concentration data were regressed against time in days. Output from the NLIN procedure included the first-order dissipation rate constant (*k*) and upper and lower confidence intervals. These values were converted into days to 50% (DT₅₀) using the equation (1)

$$DT_{50} = \ln 0.50/k$$

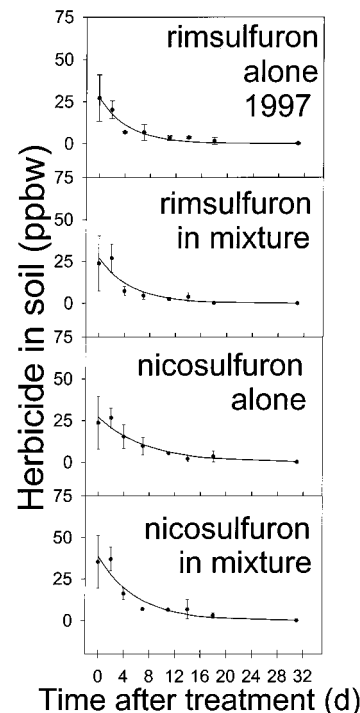
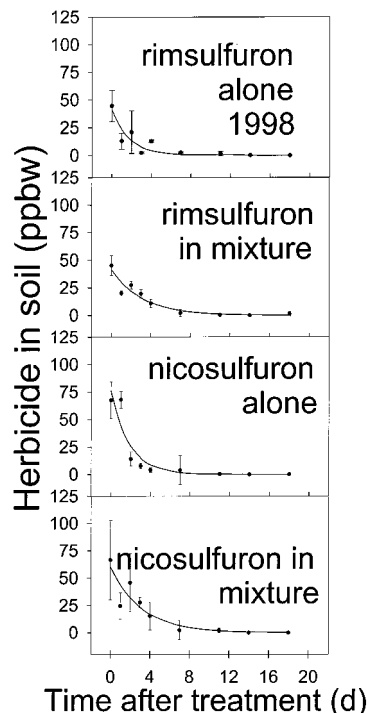
A “corrected” r² value was determined by the formula

$$r^2 = \frac{1 - (\text{residual sums of squares}/\text{corrected total sums of squares})}{1 - (\text{residual sums of squares}/\text{total sums of squares})}$$

This is a conservative approach because two standard deviations are given for each rate constant (22).

RESULTS AND DISCUSSION

Field Study. The observed half-lives of both herbicides in mixture were equivalent to the dissipation rate when applied alone (Table 2). In 1997, nicosulfuron applied alone and in a

**Figure 2.** Concentration of nicosulfuron and rimsulfuron alone and in mixture during July 1997 in surface soil (0–10 cm). Data points represent means of four measurements ± standard error. Results of first-order regression analysis are in Table 2.**Figure 3.** Concentration of nicosulfuron and rimsulfuron alone and in mixture during July 1998 in surface soil (0–8 cm). Data points represent means of four measurements ± standard error. Results of first-order regression analysis are in Table 2.

mixture with rimsulfuron had a DT₅₀ of 5.3 and 4.2 days, respectively (Figure 2). Rimsulfuron half-lives in 1997 were 3.1 and 3.5 days alone and in mixture, respectively. In 1998, there were also no differences in dissipation rate between the sulfonylureas applied alone and in mixture (Figure 3; Table 2). Herbicide concentration decreased over time; however, initial

Table 3. Daily Precipitation (Centimeters) prior to and during Sampling Period in 1997 and 1998 at Field Location

DAT	1997	1998	DAT	1997	1998
-5	0.00	0.00	14	0.00	0.00
-4	0.00	0.00	15	0.00	0.00
-3	1.32	0.00	16	0.05	0.00
-2	0.00	0.00	17	0.00	0.00
-1	0.48	0.00	18	0.00	0.00
0	0.30	0.00	19	0.00	0.00
1	3.66	1.24	20	0.00	0.00
2	0.00	0.00	21	0.00	0.00
3	0.00	0.97	22	0.00	0.00
4	0.00	0.00	23	6.86	0.76
5	1.78	0.00	24	0.10	3.15
6	0.00	0.00	25	0.00	2.77
7	0.00	0.00	26	0.00	0.08
8	0.00	0.00	27	0.00	0.00
9	0.00	0.05	28	0.00	2.34
10	1.78	2.08	29	0.00	0.00
11	0.00	0.00	30	0.00	0.00
12	0.00	0.00	31	0.00	0.79
13	0.00	0.00			
			total	16.32	14.22

Table 4. Maximum and Minimum Daily Temperatures (Degrees Centigrade) prior to and during Sampling Period in 1997 and 1998 at Field Location

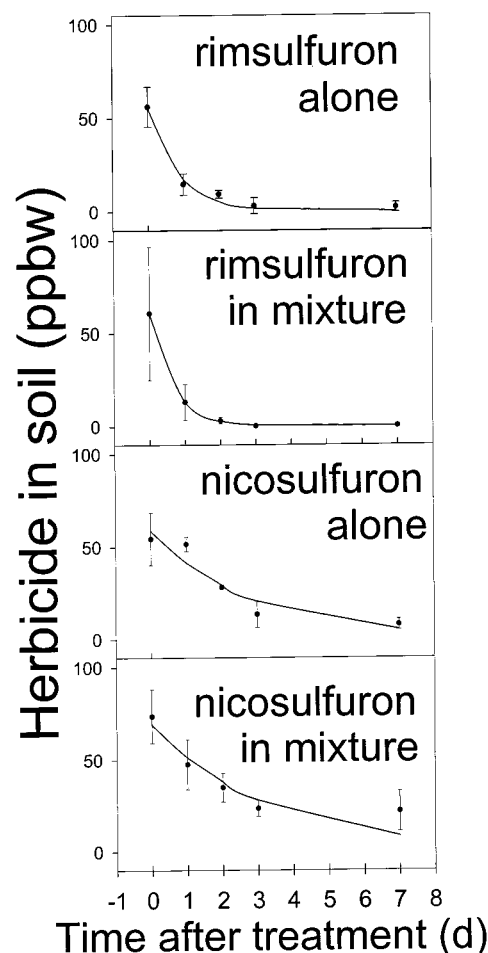
DAT	1997		1998		DAT	1997		1998	
	max	min	max	min		max	min	max	min
-5	32.2	19.4	32.2	17.8	14	31.7	20.0	30.0	20.0
-4	32.2	20.6	32.2	17.8	15	33.3	19.4	27.8	17.8
-3	30.6	21.1	32.2	20.0	16	33.3	19.4	31.1	18.9
-2	30.0	19.4	32.2	20.0	17	32.2	19.4	28.9	19.4
-1	30.0	19.4	32.2	20.6	18	32.8	18.3	30.6	16.1
0	28.9	20.6	31.1	21.7	19	33.3	19.4	30.6	18.3
1	30.0	21.1	30.6	16.7	20	34.4	20.0	32.8	17.8
2	30.0	19.4	28.3	16.1	21	34.4	20.6	31.1	18.3
3	32.8	20.0	28.3	16.7	22	33.9	22.2	32.8	18.9
4	35.0	18.3	29.4	18.9	23	33.9	21.1	30.6	19.4
5	32.2	18.9	30.0	20.0	24	32.8	20.6	28.9	19.4
6	26.7	14.4	30.0	17.8	25	32.2	19.4	28.3	20.0
7	28.3	16.1	30.6	18.9	26	32.2	19.4	28.3	18.3
8	27.8	16.1	30.6	20.0	27	32.8	20.0	27.8	18.9
9	31.1	17.8	27.8	21.1	28	31.7	22.2	27.2	18.9
10	28.3	18.3	30.0	19.4	29	33.9	20.0	29.4	20.6
11	28.9	17.2	29.4	18.3	30	31.1	20.6	29.4	20.6
12	30.6	19.4	29.4	17.8	31	28.9	17.2	27.2	20.0
13	31.1	19.4	31.1	19.4					

herbicide concentration differed by year. This was attributed to a difference in sampling technique with deeper samples being taken in 1997, which resulted in more dilution due to greater soil volume. However, all samples were taken to a consistent depth throughout a given year. Both herbicides dissipated quickly in the field and would cause minimal problems for crop rotation.

Rimsulfuron degradation has been reported to be more rapid in warm, moist, light-textured soils with low pH (13). In both years, rainfall occurred within 12 h of application to favor microbial degradation (Table 3). Soil pH was 5.7, and this would favor a combination of chemical hydrolysis and microbial degradation (4). Temperature throughout the field study interval was >16 °C (Table 4). Another possible explanation may be that some herbicide leached below the zone of soil sampling, although this was not indicated in previous research (9, 11, 14). Under different environmental conditions, nicosulfuron and rimsulfuron dissipations may differ. However, in soils with similar properties, rapid dissipation of these herbicides would be expected.

Table 5. First-Order Dissipation Rate Constants (k), Calculated Half-Lives (DT_{50}), and 95% Confidence Intervals (CI) for Herbicides in Surface Soil from Soil Fortification Experiments

herbicide treatment	r^2	k , day ⁻¹	DT_{50} , days	CI, days
nicosulfuron alone	0.93	0.34	2.0	1.2–5.9
nicosulfuron mix	0.92	0.30	2.3	1.4–7.0
rimsulfuron alone	0.98	1.15	0.6	0.5–0.9
rimsulfuron mix	0.99	1.53	0.45	0.4–0.5

**Figure 4.** Concentration of nicosulfuron and rimsulfuron alone and in mixture in soil under controlled conditions. Data points represent means of six measurements \pm standard error. Results of first-order regression analysis are in Table 5.

Laboratory Study. Dissipation rates between herbicides did not differ either alone or in mixture (Table 5). All herbicide dissipation patterns in the laboratory experiment followed first-order kinetics, with all r^2 values >0.92. With pH, temperature, and moisture held constant, there were no differences in herbicide half-lives when applied alone or simultaneously. Herbicide concentrations decreased rapidly with time (Figure 4). Herbicide dissipation in the soil was more rapid in the laboratory compared to the field, although neither herbicide was persistent ($DT_{50} < 3$ days). Rimsulfuron dissipation was more rapid than nicosulfuron dissipation (Table 5). Temperature differences between laboratory and field experiments may explain observed differences with rimsulfuron (Table 4).

The presence of nicosulfuron did not influence the degradation of rimsulfuron and rimsulfuron presence did not influence nicosulfuron degradation. Although biological sensitivity of rotational crops and weedy plant species to the examined

herbicides is a factor, this research indicated minimal risk of carry-over to subsequent rotational crops and minimal residual weed control from these herbicides when applied to a Sequatchie silt loam soil under ambient climatic conditions in Tennessee.

ABBREVIATIONS USED

DAT, days after treatment; LDPE, low-density polyethylene; DT₅₀, days to 50% herbicide loss or calculated half-life.

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